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博士学位论文

聚苯乙烯负载型和锌-腺嘌呤骨架型材料的构建及其催化性能研究

Construction of Polystyrene-supported and Zinc-Adeninate Framework Materials and Investigation of Their Catalytic Performance

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摘　要

在倡导可持续发展和绿色能源化学背景下，工业催化朝着少分离、低能耗的多相催化发展，同时也向高附加值的精细化工发展。特别是面对石化枯竭和能源结构过于单一化的隐忧，催化一方面积极开拓合成新能源化学品的反应，如生物质转化等，另一方面也致力于从碳一化学的角度拓展碳链增长的反应丰富合成路线，为可能的能源危机做技术储备。其中，CO2作为丰富安全的碳资源，在温室效应等环境问题日益恶化的情况下更加受关注，对其合理地催化转化将有助实现全球碳循环并丰富能源结构。

本文以工业应用较成熟的聚苯乙烯型离子交换树脂为起点，以低选择性的甲醛丙酮羟醛缩合制较不稳定化合物丁酮醇的碳链增长碱催化反应为探针入手。尝试表面活性剂的引入和设计索氏提取反应，发现提高反应速率和实现中间目标产物丁酮醇和催化剂的分离，都能有效避免深度反应的进行，产率得以提高。进一步地，对商业化树脂进行改性，主要通过以下途径：（1）对叔胺树脂通过Menshutkin氮烷基化反应在叔胺氮上引入长碳链；（2）在叔胺基团存在下，对聚苯乙烯骨架苯环上通过Friedel -Crafts烷基化反应引入长链酰基，并接着通过Menshutkin反应在叔胺上引入长碳链，从而构成双长碳链。催化结果表明，通过途径（1），丁酮醇产率由44.3%上升超过50%；而通过途径（2），产率达到71.3%。气质分析显示，弱碱叔胺相对于强碱季铵树脂，其催化副产物减少，而改良后的树脂其催化副产物进一步下降。CO2-TPD和HCHO-TPD表明，随着吸附分子尺寸的增大，在改性树脂上的吸附量明显下降，这预示着从高分子微观角度而言，长链的引入有效提高高分子内部位阻效应，使尺寸较大的丁酮醇更易从活性位点脱附下来，从而提高其选择性。

然而，上述接枝离子交换树脂受胺基影响，其热稳定性较差。我们引入含氮杂环负载于聚苯乙烯上，有效提高其热稳定性达近400℃。该材料在CO2合成环状碳酸酯上是一种高效的多相催化材料。烷基胺和卤代锌的引入均有利于活性的提高，但热稳定性因此有所下降。考察不同氮杂环对活性的影响，五元氮杂环的活性明显高于六元环。进而，通过衰减全反射傅里叶变换红外和核磁共振分析，考察产物碳酸丙烯酯（PC）和催化活性基团咪唑环（Im）的相互作用，从反应终态间接地反向推导

催化过程，提出一种可行的反应机理。碳酸丙烯酯和咪唑环均是平面五元环结构，从电子诱导效应而言，PC上的O-C-O结构正好与Im上含孤对电子的N-C=N配对。并进一步推测，在咪唑环上实现了CO2和环氧丙烷（PO）的吸附、以及PO开环和CO2的活化插入过程，相当于两个分子在咪唑环上的催化组装过程。催化结果表明，转化率达99.7%，选择性99.6%， TON达204以上。并且重复利用四次未发现活性有明显下降。

进一步地，高聚物负载量相对有限，对大宗CO2转化略显吃力，于是我们引入具有高比表面、富氮的金属有机骨架材料锌-腺嘌呤-异烟酸（Zn-Ad-Int）的构建。尝试晶体生长温度、锌盐中阴离子变化、阳离子镧掺杂、烷基胺接枝和KI添加等手段，了解到亲核试剂对PO的进攻、开环和CO2的插入加成影响重大。从而引入ZnX2-Ad-Int的构建，卤素对反应活性的影响依次有Cl-<Br-<I-，这在于碘离子既有较好的亲核性又有很好的离去性。该材料耐热性达280℃，重复利用后活性保持在96.7%左右。值得一提的是，水介质生长的ZnX2-Ad-Int-H2O热稳定性高达近400℃。

前面，我们更多的是着眼于催化材料，而最后一章则从反应入手。为了拓展CO2的催化转化应用，我们从CO2和甲醇直接合成碳酸二甲酯（DMC）研究起，通过引入疏水性的骨架材料——聚苯乙烯和金属有机骨架（MOFs），一定程度上促进了DMC的生成。相比于Zn-W-Mim，疏水性骨架Zn(bdc)(ted)0.5对铜基催化剂的负载更有利于DMC的生成。另外，在高温高压酸碱环境下，骨架材料会有一定程度的降解。苯环的活化生成苯甲酸甲酯和水等也进一步抑制了竞争反应DMC的生成。而聚苯乙烯的降解又为该反应注入了新的意义。另一方面，通过对CO2转化中较难实现反应的摸索，我们有机会将聚苯乙烯负载和金属有机骨架材料两者的一些改性手段应用其中，如不同配体的负载、金属螯合、疏水性MOFs的应用和杂化型MOFs的尝试等等。

关键词：固体碱；离子交换树脂；聚苯乙烯；羟醛缩合；丁酮醇；氮杂环；二氧化碳；碳酸丙烯酯；腺嘌呤；金属有机骨架；碳酸二甲酯

Abstract

Under the wake of the sustainable development and green energy chemistry, industrial catalysis parades towards the heterogeneous catalysis with less separation and low energy consumption, as well as the fine chemical industry with high additional value. To relieve the hidden threat and panics of exhausting fossil energy and the limited energy options, researches on catalysis concentrate efforts on the reactions exploring for new energy chemicals such as biomass conversion, as well as on C1 chemistry to expand carbon chain elongation reactions for riching synthetic routes, targeting to have preparations for the potential energy crisis. Thereinto, as a rich and safe carbon resource, CO2 attracts people's attentions for climatic issues such as the greenhouse effect. CO2 catalytic conversion constitutes a key link of the global carbon cycle, and can also improve the energy structure.

This work begins with a commercial polystyrene type ion exchange resin, and takes the aldol condensation of formaldehyde and acetone as the probe reaction, which is a carbon elongation reaction with less stable products and low selectivity. Surfactant and Soxhlet extraction are associated to improve the yield. It is inferred that both the improvement of reaction rate and the separation of the medium product 4-hydroxy-2-butanone from catalysts could reduce the side reactions, thus increased the yield. Subsequently, two types of modifications for resins are carried out: (1) one long alkyl chain on the copolymer tertiary amine via Menshutkin N-alkylation reaction; (2) two long alkyl chains: one on polystyrene aromatic ring through Friedel–Crafts alkylation under the circumstance of tertiary amine and the other on the copolymer tertiary amine using the Menshutkin N-alkylation reaction. The reaction results indicate that the yield for 4-hydroxy-2-butanone can be increased from 44.3% to around 50% for the resin with one long alkyl chain (the first case), while this can achieve up to 71.3% for the resin with

two long alkyl chains. The GC-MS analysis shows that the weaker alkali tertiary amine resin has fewer by-products than stronger alkali quaternary amine resin, and the content of by-products of modified resin decreases sharply. CO2-TPD and HCHO-TPD prove that the adsorbing capacities on the modified resins are decreasing with the size of the adsorbed molecule increasing. It indicates that adding long chain effectively improves polymer internal steric effect in the view of the polymer microstructure, then the bigger molecular 4-hydroxy-2-butanone is more likely to desorb from the active sites, thus the selectivity is improved.

However, the thermal stability of ion exchange resins is weakening by adding amine groups. N-heterocycles are introduced to load on polystyrene and the thermal stability can reach approximately 400 with such grafting. They are proved to be an efficient heterogeneous catalyst for converting CO2 into cyclic carbonate. The introduction of alkyl amines and zinc halides can both boost the activity, but it decreases the thermostability of the catalysts. A series of N-heterocycles are chose to catalyze CO2 cycloaddition. Five-membered heterocyclic compounds exhibit higher activities than six-membered ones. The interaction between the product propylene carbonate (PC) and the catalytic active group imidazole ring (Im) is studied through ATR-IR and NMR. Then, the catalytic process is inferred backward from the final state of the reaction. PC and Im are both perfectly planar with a five-membered ring structure. Based on the electron induced effect, the O-C-O of PC can match with the N-C=N of imidazole, which owns unpaired nitrogens. A plausible mechanism is proposed. CO2 and PO are adsorbed on an imidazole ring, then subsequently with the ring-opening of PO and the activation and insertion of CO2 into PO, just like a catalytic assembly process of two moleculars on one imidazole ring. Finally, the yield reaches 99.7%, with the selectivity 99.6% and TON above 204. The catalyst can be reused over four times without appreciable loss of catalytic activity.

Further, bulk CO2 conversion seems to be slightly tired due to the low loading of

polymer-supported materials and the Metal-Organic Frameworks (MOFs) may overcome such difficulty based on the current advantages. As a part of this work, a Zn-Ad-Int framework is constructed with high surface area and nitrogen-rich. The effects of MOF components on CO2 conversion are studied, including growth temperature, different zinc salts, Lanthanum embedded, alkyl amine grafting and KI addition. We find that the attack of nucleophilic reagent on PO is a key point for PO ring-opening and CO2 inserting. Then, we focus on the construction of ZnX2-Ad-Int (X=Cl, Br, I). The activity increases as followed: Cl-<Br-<I-, which is because I- owns a good nucleophilic ability, as well as a good leaving ability. Their thermostability can reach 280, and the recycling activity can keep at about 96.7%. It is worth to mention that the thermal stability of ZnX2-Ad-Int-H2O can reach approximately 400.

Prior to this, we shift our attention onto the reaction in the last chapter, as focusing on catalytic materials previously. For expanding CO2 catalytic application, we start our new research target on the direct synthesis of DMC from CO2 and methanol. The introduction of hydrophobic frameworks, such as polystyrene and MOFs, could promote DMC yield. Compared with Zn-W-Mim, Cu embeds into hydrophobic Zn(bdc)(ted)0.5 framework is more conducive to yield DMC. Moreover, under the acid and alkali circumstance with high temperature and high pressure, both the two frameworks, polystyrene and Zn(bdc)(ted)0.5 suffer with degradation. The activation of benzene ring to produce methyl benzoate and water may further suppress DMC yield. On the other hand, it also brings a new significance on the reclamation of waste polystyrene. In addition, during the exploration for difficult CO2 conversion reactions, we practice a series of modification methods on polystyrene-supported materials and MOFs, such as different ligands loading, metal chelation, hydrophobic MOF and hybrid framework applications.

Keywords: solid base; ion exchange resin; polystyrene; aldol condensation; 4-

hydro-2-butanone; N-heterocycle; carbon dioxide; propylene carbonate; adenine; metal-organic framework (MOF); dimethyl carbonate.

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